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A PRECISION CRYOSTAT WITH AUTOMATIC TEMPERATURE REGULATION

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ABSTRACT

A cryostat, which is an apparatus for maintaining constant low temperatures, has been designed and constructed for use in the temperature range from 0° to −170° C. In this range the temperature can be easily adjusted to any desired value and can be automatically maintained constant to within 0.001° C. It can also be used for maintaining temperatures at the boiling points of liquid oxygen, nitrogen, or air.

The cryostat bath is contained in a double walled cylindrical glass vessel surrounded by liquid air, the amount of refrigeration being controlled by varying the gas pressure between the walls. The cryostat liquid is circulated by a propeller in the bottom of a bakelite tube centrally located in the double walled vessel. The liquid is pumped up the inside of the bakelite tube, which is the constant temperature region of the cryostat, and down on the outside. By operating the propeller through a system of gears driven by a shaft on the outside of the bakelite tube, the whole space inside is available for experimental apparatus. Constant temperature is automatically maintained by a photo-electric cell thermo regulator which controls the current through a heating coil wound on the outside of the bakelite tube. Adequate circulation of the liquid, suitable heat insulation, and symmetrical distribution of the parts assure great uniformity of temperature. Nonflammable mixtures of liquids are used for the cryostat bath between 0° and −140° C. For lower temperatures liquid propane is used.

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I. INTRODUCTION

It frequently happens that the accuracy of the determination of a physical property is limited by the temperature variations of the supposedly constant temperature space in which the property is being measured. An apparatus has been developed which will automatically maintain any temperature, from 0° down to −170° C., constant and uniform to within 0.001° C. On account of limitations of temperature measurements and other factors this constancy and uniformity is as good as is required for practically all work in this range of temperatures.

Isolated temperatures can be maintained by substances undergoing a change of state at atmospheric pressure, and some limited ranges can be covered by controlling the pressure over boiling liquids.[1]

[1] K. Onnes, Leiden Communications Nos. 83, 94, and 123a.

It is, however, not only convenient, but for some work necessary that the temperature be adjustable to any desired point and be maintained constant automatically. For the interval near room temperatures this is relatively easy, but at higher and lower temperatures the problem becomes increasingly difficult.

Probably the most satisfactory and economical form of apparatus for use at low temperatures is a stirred liquid bath. Since liquids have rather high heat capacities, and, if well stirred will transfer heat quickly, they are well suited for maintaining constant and uniform temperatures. In providing refrigeration for the bath it is advantageous to have the heat leave the bath through a large surface, and to keep the refrigeration as constant as possible. Exposing a large cooling surface to the liquid reduces the temperature difference required to transfer a given amount of refrigeration, thereby making it possible to prevent the existence of cold spots in the bath which may occur if the refrigeration has to be transmitted through a small surface with a large temperature difference.

Since electric heating can be easily controlled through the action of a relay, the most convenient procedure for obtaining automatic temperature regulations is to provide a slight excess of refrigeration and to compensate for the excess with electric heating controlled by some thermosensitive device. The excess need be no greater than the combined maximum variations (1) of the refrigeration, (2) of the heat leakage into the bath from the warm exterior, and (3) of the energy dissipation in the bath. In order to obtain great constancy of temperature, these variations must be made small.

The constancy of temperature attainable also depends upon other factors which influence the response of the regulating device. These factors may be summarized briefly as follows: (1) Thermal lag in the thermosensitive element of the regulator, (2) time lags in the response of the regulator to changes in its thermosensitive element, (3) thermal lags in the heating and cooling elements, and (4) a time-lag effect which depends upon the velocity of circulation of the liquid and the relative positions of the heating and cooling surfaces with respect to the thermosensitive element. It is important that the temperature be constant with respect to time, but it is equally important that the temperature be uniform throughout the region of the bath in which the experimental apparatus is placed.

With these principles in mind a cryostat was designed and constructed for use with liquid air as the refrigerant. The refrigeration was applied and controlled by a method previously used by Rothe [2] and by Keyes, Taylor, and Smith,[3] namely, by surrounding the Dewar flask which holds the constant temperature bath with another Dewar filled with liquid air and controlling the refrigeration by varying the gas pressure between the two walls of the inner Dewar which is connected to a vacuum system. This method was selected in preference to other methods which might have been used because the refrigeration can be easily controlled, and by maintaining a constant liquid air level in the outer Dewar, which can be done automatically, the refrigeration remains very constant without further attention.

[2] Rothe, Zeitschrift für Instrumentendunde, **22**, pp. 14–21; 1902.
[3] Keyes, Taylor, and Smith, J. Math. & Phys. M. I. T., **1**, p. 211; 1922.

II. DESCRIPTION OF THE CRYOSTAT

A vertical section of the cryostat is shown to scale in Figure 1. It consists of an inner Dewar flask *D*, which contains the bath, surrounded by liquid air in the outer Dewar flask *C*. The inner flask is supported upon the metal ring *G*, which is connected by three copper-nickel alloy tubes *E* to the top plate *A*. The outer flask is supported from below on a pedestal which can be raised or lowered.

The refrigeration is controlled by varying the gas pressure between the walls of the inner Dewar *D*, which is connected by means of the side tube *M* to a vacuum system shown at *A* in Figure 2. In order to thermostat the cryostat at temperatures below −160° C., hydrogen or helium is used between the walls. Very low pressures, or high vacua, requiring the use of a mercury diffusion pump are necessary for maintaining temperatures near 0° C. The pressure for maintaining any temperature is not critical. Excess refrigeration resulting from a higher pressure than is needed to maintain a temperature can be compensated for with a supply of heat generated by an electric current flowing through a heating coil immersed in the cryostat bath. The automatic thermoregulator compensates for small changes in the refrigeration, heating current supply, and the flow of heat into the cryostat from the outside. Because the stirring is vigorous, and the heating and cooling surfaces are near each other, compensating for excess refrigeration with electrical heating does not affect either the constancy or the uniformity of the temperature. A considerable change of temperature can be easily and quickly effected by varying the current through the heating coil in the liquid bath without the necessity of changing the pressure between the walls of the Dewar. Gas pressures between the walls of the inner Dewar are maintained by the use of a stopcock or mercury cut-off.

As far as possible, the nonflammable mixtures recommended by C. W. Kanolt [4] are used for the cryostat bath liquid. For temperatures down to −75° C. the liquid used is the eutectic mixutre of carbon tetrachloride and chloroform (49.4 per cent, by weight, of CCl_4 and 50.6 per cent of $CHCl_3$), which has a freezing point of −81.4° C. as determined by Kanolt. For temperatures between −75° and −140° C. a 5-component mixture containing 14.5 per cent of chloroform, 25.3 per cent of methylene chloride, 33.4 per cent of ethyl bromide, 10.4 per cent of transdichloroethylene, and 16.4 per cent of trichloroethylene was used. This mixture freezes at about −150° C.

At temperatures below −140° C. the nonflammable liquids become so viscous that they can not be readily stirred. For this lower range the liquid used is commercial propane, which is sold for domestic gas heating under various trade names. The propane, which is a gas at atmospheric pressure and room temperature, is compressed into steel cylinders and liquefied at a pressure of about 150 lbs./in.^{2} It can be liquefied at atmospheric pressure by running it through a copper coil immersed in liquid air. Its boiling point is about −44° C., and it remains liquid down to liquid air temperatures. It is, however, highly flammable and its use is attended with some danger. It is recommended that pyrex glass or metal Dewars be used, and precautions be taken to prevent the mixing of the liquid air and

[4] C. W. Kanolt, B. S. Sci. Paper No. 520; March, 1926.

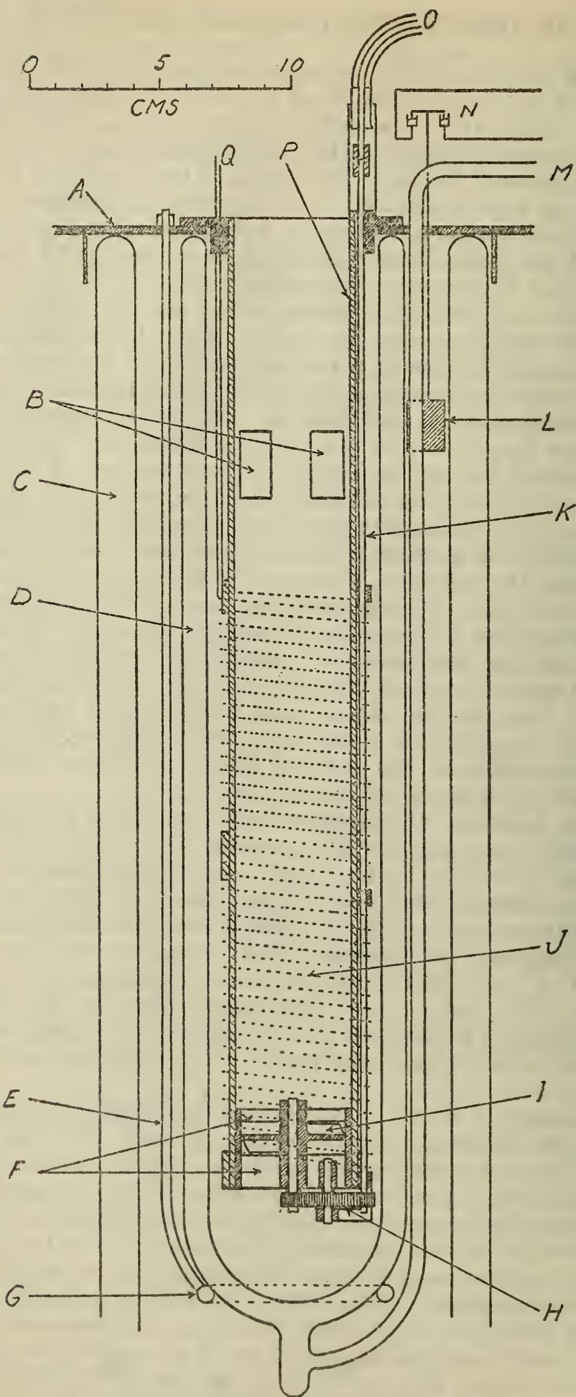

FIGURE 1.—*Vertical section of the cryostat*

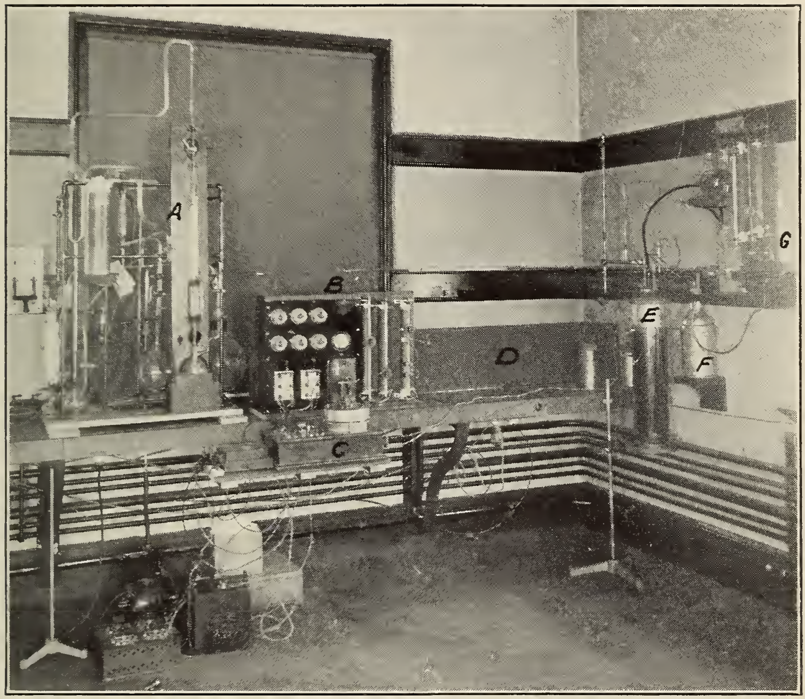

FIGURE 2.—*The cryostat and accessory apparatus*

propane in case the Dewars break. If liquid nitrogen is available, it should be used instead of liquid air for the refrigerating liquid.

The cryostat bath liquid is circulated by a propeller *I* (see fig. 1) which revolves in the bottom of a bakelite tube *P* in such a direction that the liquid passes up through the inside of the bakelite tube and down on the outside. To permit this circulation, four windows *B*, 2.5 cm long, were cut in the bakelite tube. In order that the liquid at the surface of the bath shall be drawn away as its temperature is changed by the heat conducted in from the warm exterior, the liquid level is kept below the top of the windows. The propeller is driven at a speed of about 400 revolutions per minute by a system of three gears *H* driven by a shaft *K* connecting to a motor through a flexible shaft *O*. Three vertical vanes *F* support the bearings for the propeller and idler gear shafts and improve the circulation by preventing a whirling motion of the liquid above and below the propeller. Although gears made of brass running against stainless steel were found satisfactory, a combination of metal against bakelite was found to be quieter.

In the beginning trouble was experienced with a wearing away of the bearings and the shafts rotating in them, because the bearing surfaces were not lubricated, and the cryostat bath liquids attacked the bearing surfaces and formed substances on them that acted as abrasives. Several different combinations of metals were tried, and Monel metal shafts turning in a high lead (85 per cent) babbitt were used. New bearings are first "run in" with a good lubricating oil, cleaned with alcohol and rubbed with dry graphite before assembling for use.

The stirring mechanism can be quickly and easily taken apart and reassembled, which is desirable, as this must be done occasionally when ice collects in the gears.

The heating coil *J*, shown by dotted lines, is made of No. 22 B. & S. gage Nichrome wire and has a resistance of about 30 ohms. The bare wire is wound bifilarly on a form which fits on the stirrer tube *P*, its ends terminating at the top in the leads *Q* which connect to the thermoregulator. The form is made from a bakelite tube by cutting wide slots in its walls, leaving six strips about 1 cm. wide and supporting rings at the ends and center. Notches were cut in the strips to keep the wire from slipping. Lag in the heater is reduced by this support and manner of winding which separates the heater coil from the stirring tube and allows the bath liquid to circulate on all sides of the wire. By distributing the heater instead of concentrating it in a small part of the bath, the existence of hot spots in the cryostat is prevented. It is advantageous in compensating for a large excess of refrigeration, over that needed to maintain a desired temperature, to have the heating surface close to and distributed over the refrigerating surface. This, together with the vigorous stirring, makes it possible to operate under such conditions without affecting the constancy or uniformity of temperature in the constant temperature space.

The level of the liquid air in the outer Dewar is kept constant by means of an automatic constant level device.[5] The float *L* operates through the contacts *N* and relay *R*, a solenoid-operated valve *V*

[5] See Scott and Brickwedde, An Automatic Constant Level Device, Rev. Sci. Inst. (in press).

opening and closing a low-pressure air supply to the flask F, as shown diagrammatically in Figure 3. Due to the splashing and boiling of the liquid air, only a small amount is added each time the valve operates, and the level does not vary more than 1 or 2 millimeters.

The thermoregulator used is similar to a thermoregulator previously described by Southard and Andrews [6] and is diagrammatically represented in Figure 4. The thermosensitive sensitive element of the regulator is a 10-junction thermocouple which is represented by a

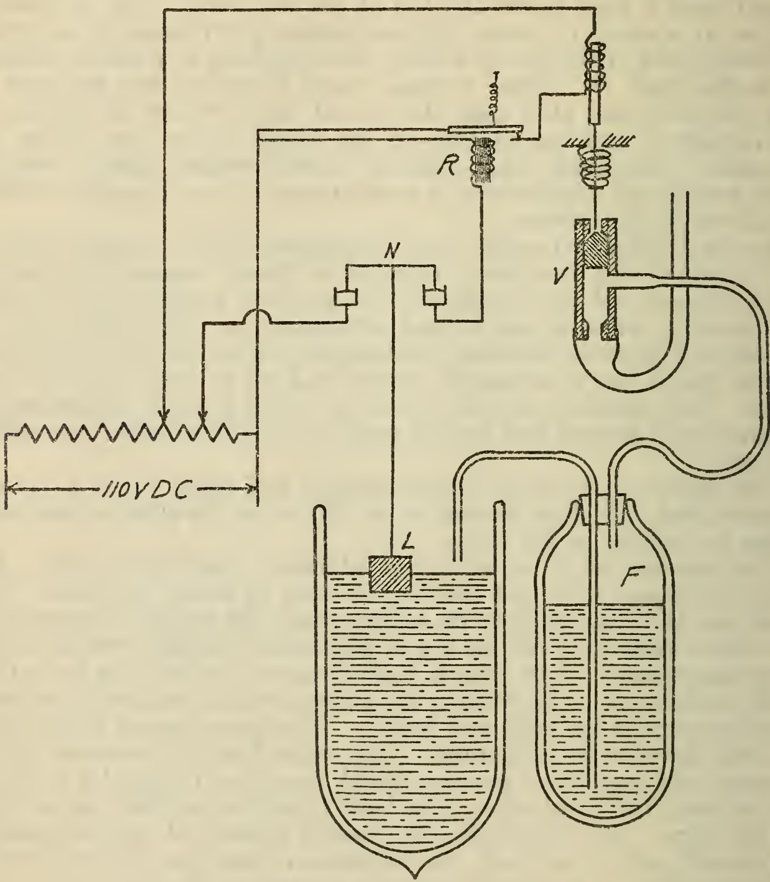

FIGURE 3.—*Diagram of the constant level device*

simple thermocouple T. One set of junctions spread out fanlike is placed just above the propeller, the external junctions being in an ice bath. The two leads of the multiple junction thermocouple are connected to a Leeds & Northrup, type K potentiometer P which may be set to the electromotive force of the thermocouple corresponding to the desired temperature. The potentiometer is balanced and used in much the same manner it would be used to measure the temperature of the cryostat with a thermocouple. Although a potentiometer is

[6] Southard and Andrews, J. Frank. Inst., **207**, pp. 323-339; 1929.

convenient, it can be replaced by an inexpensive circuit made up of adjustable resistances which will perform the same function as the potentiometer; that is, of balancing the electromotive force of the 10-junction thermocouple with a potential difference across a resistance through which a current flows. To the potentiometer is connected a Leeds & Northrup, type R galvanometer G having a resistance of about 40 ohms. An image of the straight filament of a lamp L is formed by the concave galvanometer mirror upon the knife-edge of a screen K placed in front of and to the side of the photo-electric cell so that when the temperature in the cryostat falls, and the galvanometer mirror is deflected, the image of the lamp filament will move across the knife-edge onto the sensitive plate of the photo-electric cell, whereas when the temperature rises, the image of the lamp filament will cross the

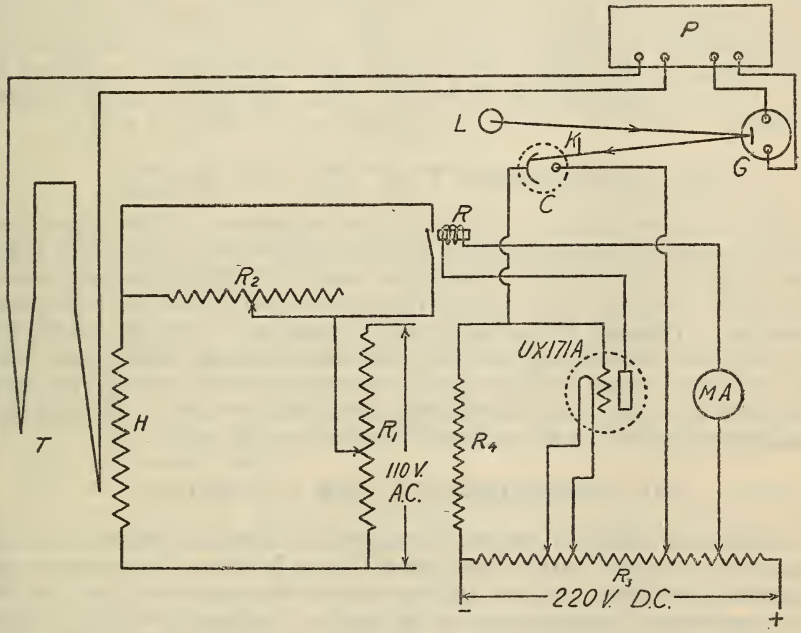

Figure 4.—*Diagram of the thermoregulator circuit*

knife-edge onto the screen which will shade the sensitive plate of the cell from the light of the lamp. The photo-electric cell used is of the gas filled, caesium coated plate type. The photo-electric current generated when the light falls on the cell is amplified by means of a single UX No. 171A radio tube, to the grid of which the cathode of the photo-electric cell is connected as shown in the diagram. The various voltages necessary for the photo-electric cell and amplifier are obtained from a slide wire rheostat R_3 of about 400 ohms, equipped with auxiliary sliders, and connected across a 220 volt d. c. supply. The optimum voltages will vary with the cell and amplifier. A potential difference of 135 volts was used in the plate circuit of the amplifier and 70 volts in the photo-electric cell circuit. The grid of the amplifier is given a negative bias of about 30 volts through the resistance

R_4 of 40 megohms which is made up of four 10-megohm grid leaks in series.

In the plate circuit of the amplifier tube is a milliameter (*MA*) and a 2,000-ohm relay *R* which opens and closes a shunt circuit around the 50-ohm rheostat R_2 which is connected in series with the heating coil *H* in the cryostat bath. The voltage applied to this series circuit may be varied from 0 to 110 volts by the rheostat R_1, having a resistance of 400 ohms. The photo-electric cell and amplifier circuit are carefully insulated and the grid leaks and mountings are coated with shellac to make them less affected by humidity. These precautions are necessary for satisfactory operation.

As an alternative to the use of the thermocouple, a coil of platinum wire was used as the thermosensitive element of the regulator by using the coil as one arm of a Wheatstone bridge circuit. The coil, which has a resistance of about 20 ohms at 0° C., was made by winding bare platinum wire on a light frame of mica, and in order to compensate for changes in resistance of the leads, three leads were connected to the coil as is done in the three lead, Siemens type resistance thermometer. For this purpose the bridge ratio should be unity.

III. ARRANGEMENT OF THE APPARATUS

The photograph (fig. 2) shows the arrangement of the apparatus. *E* is the cryostat proper with the stirrer motor above and to the right. The relay and valve for the constant level mechanism are shown at *G*. The flask *F* contains the supply of liquid air to replenish that in the cryostat. The box *D* contains the galvanometer and photo-electric cell with the amplifying circuit. All the electrical connections lead to the control panel *B* upon which is mounted also the thermoregulator relay and the resistances controlling the heating current. The potentiometer is shown at *C*, and the vacuum system at *A*.

IV. OPERATION OF THE CRYOSTAT

In order to cool the cryostat down to the desired temperature, the space between the walls of the inner Dewar is allowed to come to atmospheric pressure and the falling temperature observed by a platinum resistance thermometer in the bath. Cooling down from room temperature may require from one-half to two hours depending upon the temperature. When the desired temperature is almost reached, the system is evacuated until the temperature is falling very slowly; the temperature now being observed with another galvanometer which can be connected in series with the thermoregulator galvanometer *G* of Figure 4. The heater resistances are now adjusted until the steady current almost, but not quite, balances the loss of heat from the cryostat. The thermoregulator is started and its operation observed by means of the external galvanometer. Further adjustment of the heating current may be necessary to get the best operation. The difference between maximum and minimum heating current should be just enough to take care of any irregularities of heat conduction into the bath. In order to change to a higher temperature, a heavy current is sent through the heater. A rise in temperature of 20° or 30° may be effected in about 10 minutes.

V. CONSTANCY AND UNIFORMITY OF TEMPERATURE

The external galvanometer also provides an excellent means of determining the temperature variations as the thermoregulator operates. Since the regulator goes through two or three cycles per minute, the temperature oscillations are largely damped in a glass-encased resistance thermometer placed in the bath. However, by observing the deflections of the galvanometer and knowing its sensitivity and the total resistance of the circuit, as well as the thermoelectric power of the thermocouple, the temperature variations may be calculated. It was found that these variations could be made less than 0.001° C. The temperature variations in the bath, using the platinum coil as the thermosensitive element of the regulator, were determined in a similar manner and found to be of about the same magnitude. This constancy and the relatively short period of the regulator indicate that temperature lags and irregularities are very small. In observing the thermoregulator it was noticed that the image of the filament of the lamp L of Figure 4 moves less than a millimeter, corresponding to a change in the electromotive force of the thermocouple of about 0.2 microvolt.

The constancy of temperature over a long period of time depends upon the steadiness of the zero point of the thermoregulator galvanometer, and upon the maintenance of constant current in the potentiometer when the thermocouple is used as the thermosensitive element. A good galvanometer will maintain its zero point very well, so this cause of variation is unimportant. However, there is usually a steady drift in the value of the current through the potentiometer, and this should be made as small as possible by using a good battery as a source of current and insulating this battery against temperature changes. If it is necessary to keep the temperature constant to 0.001° C. for a long time, the potentiometer current or setting should be adjusted occasionally.

When using a resistance as the sensitive element of the regulator, the greatest cause of drift of the thermostating temperature is the change in resistance of the bridge with room temperature. Except when operating near 0° C., the drift is less when using the resistance coil as the thermosensitive element than when using the thermocouple. This comparison was made using manganim resistance coils in the Wheatstone bridge and a lead storage cell as the source of potentiometer current.

In order to test for uniformity of temperature, the bath was explored with a 10-junction copper constantan difference thermocouple. No differences as great as 0.001° C. could be detected in the constant temperature region.

VI. SUMMARY

A cryostat, for use in the temperature range from 0° to −170° C., has been designed and constructed which will automatically maintain a temperature constant and uniform to 0.001° C. Adequate circulation, suitable heat insulation, and a symmetrical distribution of the parts assure great uniformity of temperature. The temperature is maintained constant automatically by a photo-

electric cell thermoregulator. The design is such that it allows for a large unobstructed constant temperature space.

The cryostat is being used for the calibration and standardization of thermometers and for the investigation of the physical properties of materials at low temperatures.

WASHINGTON, January 5, 1931.

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